IS 8063 (1976): Red lead for explosive and pyrotechnic industry [CHD 26: Explosives and Pyrotechnics]

BLANK PAGE

Indian Standard

SPECIFICATION FOR RED LEAD FOR EXPLOSIVE AND PYROTECHNIC INDUSTRY

UDC 661.851.3 : 662-l/.4

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr. 6 ***October* 1976**

AMENDMENT NO. 1 MARCH 2000
TO
IS 8063:1976 SPECIFICATION FOR RED LEAD FOR EXPLOSIVE AND PYROTECHNIC INDUSTRY

(*Page* 14, *clause* **A-6.3**) — Substitute the following for the existing formula:

'Lead as $P_{b5}O_4$, percent by mass = 3.428(V_1 - V_2)'

(CHD 26)

Reprography Unit, BIS, New Delhi, India

Indian Standard
SPECIFICATION FOR RED LEAD FOR EXPLOSIVE AND PYROTECHNIC INDUSTRY

Explosives and Pyrotechnics Sectional Committee, CDC 51

Chairman	*Representing*
DR W. D. PATWARDHAN	Ministry of Defence (R & D)
Members	
CHAIRMAN, MANAGERS' COMMITTEE	Bharat Gold Mines Ltd, Oorgaum
DR A. K. CHATTERJEE	IDL Chemicals Ltd, Hyderabad
SHRI V. Y. TAMBE (*Alternate*)	
SHRI L. S. CHEEMA	Fertilizer Corporation of India Ltd (Nangal Unit), Naya Nangal
ASSISTANT SUPERINTENDENT, FCI, DHANBAD (*Alternate*)	
SHRI D. P. CHOUDHRY	National Coal Development Corporation Ltd, Ranchi
SHRI R. G. DEOLALIKAR	Directorate General of Ordnance Factories (Ministry of Defence), Calcutta
SHRI K. P. R. PILLAI (*Alternate*)	
SHRI C. M. DEOSTHALE	Directorate General of Mines Safety, Dhanbad
SHRI B. P. GUPTA	Directorate General, Border Roads, New Delhi
SHRI P. C. GUPTA	National Mineral Development Corporation Ltd, New Delhi
SHRI R. D. JINDAL	Indian Mining Federation, Calcutta
SHRI JOGINDER SINGH	Directorate General of Tachnical Development, New Delhi
SHRI JASTINDER SINGH (*Alternate*)	
SHRI S. C. KALE	Directorate General of Factory Advice Service and Labour Institutes, Bombay
SHRI R. R. KHANNA	Indian Mining Association, Calcutta
SHRI V. KRISHNAMURTHY	Central Mining Research Station (CSIR), Dhanbad
SHRI S. G. KULKARNI (*Alternate*)	
SHRI I. N. MURTY	Chief Controller of Explosives, Nagpur
SHRI K. S. SUBRAMANYAM (*Alternate*)	
SHRI K. C. NARAYANAN	Development Commissioner, Small Scale Industries, New Delhi
DR J. N. SEN	Indian Explosives Ltd, Calcutta
SHRI G. M. CHOPRA (*Alternate*)	
SHRI K. B. SENGUPTA	Associated Cement Companies Ltd, Bombay
SHRI M. M. PRABHU (*Alternate*)	

(*Continued on page* 2)

(*Continued from page* 1)

Members	*Representing*
DR S. K. SINHA	Ministry of Defence (R & D)
DR K. R. K. RAO (*Alternate*)	
DR SURJIT SINGH	Ministry of Defence (DGI)
SHRI V. SUBRAMANIAN (*Alternate*)	
DR G. M. SAXENA, Director (Chem)	Director General, ISI (*Ex-officio Member*)

Secretary

SHRI S. K. MATHUR
Deputy Director (Chem), ISI

Raw Materials Subcommittee, CDC 51 : 4

Convener	
SHRI G. M. CHOPRA	Indian Explosives Ltd, Calcutta
Members	
DR B. N. ROY (*Alternate* to Shri G. M. Chopra)	
SHRI R. G. DEOLALIKAR	Directorate General of Ordnance Factories (Ministry of Defence), Calcutta
SHRI K. M. SARAPH	Ministry of Defence (DGI)
SHRI M. G. BHAGWAT (*Alternate*)	

Indian Standard
SPECIFICATION FOR RED LEAD FOR EXPLOSIVE AND PYROTECHNIC INDUSTRY

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 10 May 1976, after the draft finalized by the Explosives and Pyrotechnics Sectional Committee had been approved by the Chemical Division Council.

0.2 Red lead is a synthetic inorganic chemical widely used in the manufacture of delay detonators in pyrotechnic formulations. The standard, IS : 57-1965* caters to the requirements of paint industry and does not serve the purpose of explosive and pyrotechnic compositions. This standard has, therefore, been formulated with a view to facilitating the procurement of standard quality red lead for use in explosive and pyrotechnic industry.

0.3 This standard contains clause **3.1** which calls for agreement between the purchaser and the supplier.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960†. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for red lead (minium) for use in explosive and pyrotechnic compositions.

2. REQUIREMENTS

2.1 Form and Condition — The material shall be in the form of reddish orange powder, free from oil, grit, dirt and other visible and extraneous impurities.

*Specification for red lead for paints and jointing purposes (*revised*).

†Rules for rounding off numerical values (*revised*).

2.2 The material shall comply with the requirements given in Table 1 when tested according to the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 4 of the table.

TABLE 1 REQUIREMENTS FOR RED LEAD FOR EXPLOSIVE AND PYROTECHNIC INDUSTRY

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST (REF TO CL No. IN APPENDIX A)
(1)	(2)	(3)	(4)
i)	Volatile matter, percent by mass, *Max*	0.05	A-2
ii)	Matter soluble in water, percent by mass, *Max*	0.3	A-3
iii)	Fineness, material retained on 45-micron IS Sieve	Nil	A-4
iv)	Average particle size, microns (by air permeability method)	1-3	A-5
v)	Assay (as Pb_3O_4), percent by mass, *Min*	97.0	A-6
vi)	Arsenic (as As_2O_3), ppm, *Max*	5	A-7
vii)	Antimony (as Sb), ppm, *Max*	20	A-7
viii)	Copper (as Cu), ppm, *Max*	20	A-7
ix)	Bismuth (as Bi), ppm, *Max*	10	A-7
x)	Iron (as Fe), ppm, *Max*	200	A-7
xi)	Zinc (as Zn), ppm, *Max*	10	A-7

3. PACKING AND MARKING

3.1 Packing — The material shall be suitably packed as agreed between the purchaser and the supplier.

3.2 Marking — The containers shall be marked legibly and indelibly with the following information:

a) Name of the material;

b) Manufacturer's name and/or his recognized trade-mark, if any;

c) Tare and net mass of the material;

d) Year of packing; and

e) Identification mark in code or otherwise to enable the batch of material to be traced from records.

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 The method of drawing representative samples of the material, the number of tests to be performed and the criteria for conformity of the material to the requirements of this specification shall be as prescribed in Appendix B.

APPENDIX A

(*Clause* 2.2)

METHODS OF TEST FOR RED LEAD FOR EXPLOSIVE AND PYROTECHNIC INDUSTRY

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960*) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF VOLATILE MATTER

A-2.1 Procedure — Heat a flat bottomed clean glass or aluminium dish, with cover, about 6 cm in diameter and 3 cm in depth in an oven at 100 ± 2°C for half an hour. Cool it in a desiccator and weigh. Place about 10 g of the material in the dish, replace the cover and weigh accurately. Uncover the dish and heat the sample and cover in an oven maintained at 100 ± 2°C for 2 hours. At the end of this period replace the cover, cool the dish and the cover in a desiccator to room temperature and weigh. Repeat the operation till constant mass is obtained.

*Specification for water, distilled quality (*revised*).

A-2.2 Calculation — Calculate the volatile matter as follows:

$$\text{Volatile matter, percentage by mass} = \frac{100 \times (M_2 - M_3)}{(M_2 - M_1)}$$

where

M_2 = mass in g of the dish and the cover with the sample taken, and

M_3 = mass in g of the dish and the cover with the sample after heating, and

M_1 = mass in g of the empty dish and the cover.

A-3. DETERMINATION OF MATTER SOLUBLE IN WATER

A-3.1 Procedure — Weigh accurately about 10 g of the material dried as prescribed under **A-2** and transfer it to a beaker. Wet it thoroughly with 95 percent by volume of ethyl alcohol or rectified spirit and then add 200 ml of water. Boil for 5 minutes. Cool the mixture to room temperature and transfer to a 250-ml volumetric flask. Make up to 250-ml with freshly boiled and cooled water. Shake and filter. Reject about 50 ml of the first portion of the filtrate. Take a 100-ml portion from the rest of the filtrate in a tared porcelain dish and evaporate to dryness on a water-bath. Dry the residue so obtained to constant mass in an oven at 100 ± 2°C.

A-3.2 Calculation — Calculate the matter soluble in water as follows:

$$\text{Matter soluble in water, percent by mass} = \frac{(M_2 - M_1) \times 2{\cdot}5 \times 100}{M}$$

where

M_2 = mass in g of the dish with the residue,

M_1 = mass in g of the empty dish, and

M = mass in g of the material taken for test.

A-4. DETERMINATION OF FINENESS

A-4.1 Procedure — Place 10 g of the material on 45-micron IS sieve and brush it gently with a 25-mm varnish brush for 15 minutes or until no further material passes through the sieve, whichever is the lesser period. Remove the sieve and weigh the portion of the sample retained on it.

A-4.2 The material shall be considered to have passed the requirement of the test if there is no residue left on the sieve.

A-5. DETERMINATION OF AVERAGE PARTICLE SIZE AND SPECIFIC SURFACE (AIR PERMEABILITY METHOD)

A-5.0 Outline of the Method — The specific surface of the powder is first measured by the time of flow of a known volume of air through a compact bed of the material. The mathematical basis for the procedure is complex and to a certain extent empirical. The mean particle diameter is then calculated from its specific surface if the particle shape is known. For spherical particles, the following formula shall be used:

$$X = \frac{6}{S}$$

where

X = mean particle diameter in cm, and

S = specific surface in cm^2/cm^3.

A-5.1 Apparatus — as shown in Fig. 1*. It consists of a graduated glass U-tube manometer, a ground glass joint supporting a stainless steel test cell and a glass tube with stopcock leading to a rubber air aspirator bulb, all mounted on a wooden support stand forming one compact portable self-contained assembly. Behind the manometer is a stainless steel mirror to permit easier reading of the graduations of the manometer and to reduce parallax. The stainless steel test cell has an inner ledge which supports a stainless steel perforated disc. The test cell can also hold a stainless steel plunger used for compacting the sample bed. The manometer contains diesel oil of known density up to a height of 12 cm from the bottom of the U-tube. The dimensions of the apparatus, the cell and the plunger are shown in Fig. 2.

A-5.1.1 Alternatively, Fischer sub-sieve sizer may also be used.

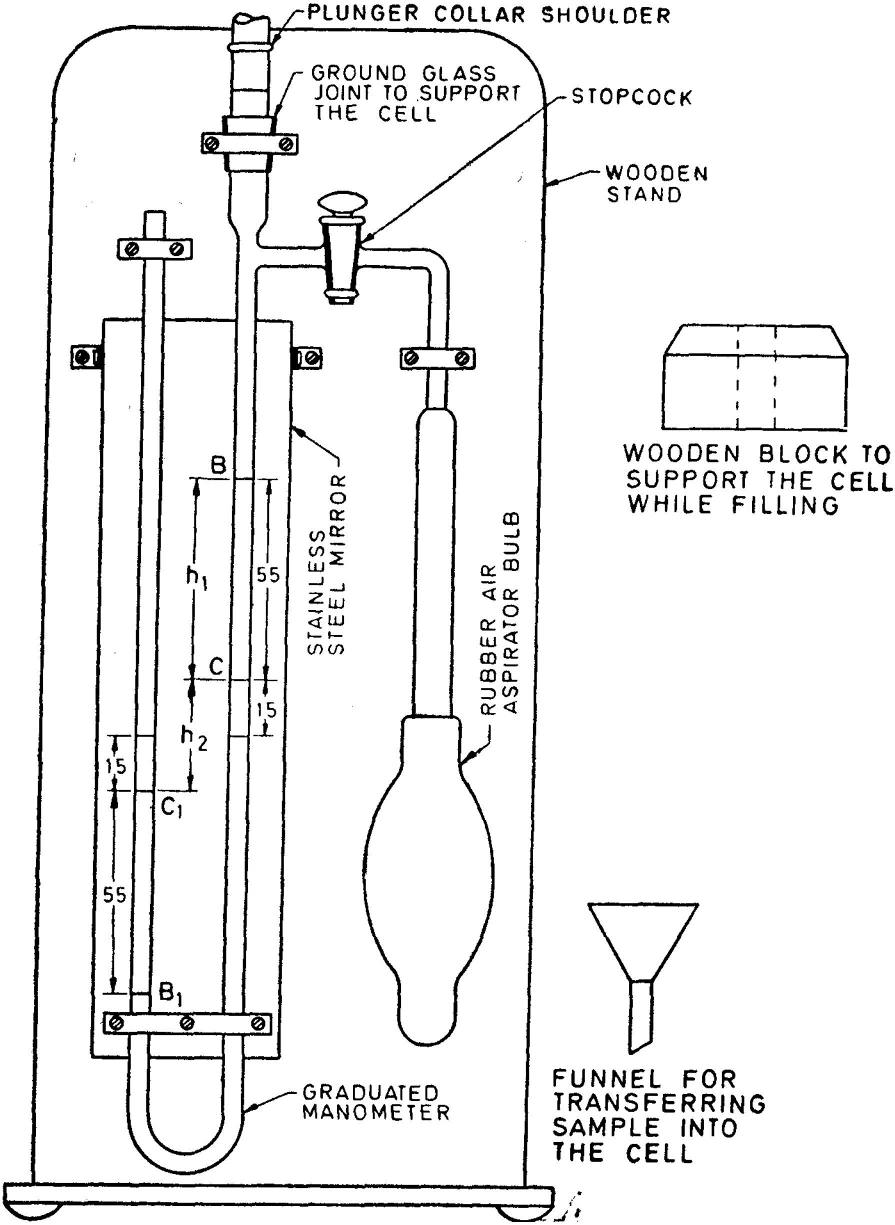
A-5.2 Determination of Constants of the Test Cell

A-5.2.1 Determine the inside diameter of the cylindrical test cell by means of slide calipers and calculate the area of the bed of powder in cm^2 (A).

A-5.2.2 Determine the depth of the cell with the perforated disc and the two filter paper discs (that will be used later in the experiment) placed on the metal perforated disc. Subtract from the cell depth, the height of the stainless steel plunger from bottom end to collar, to obtain depth of the bed of powder in cm (L).

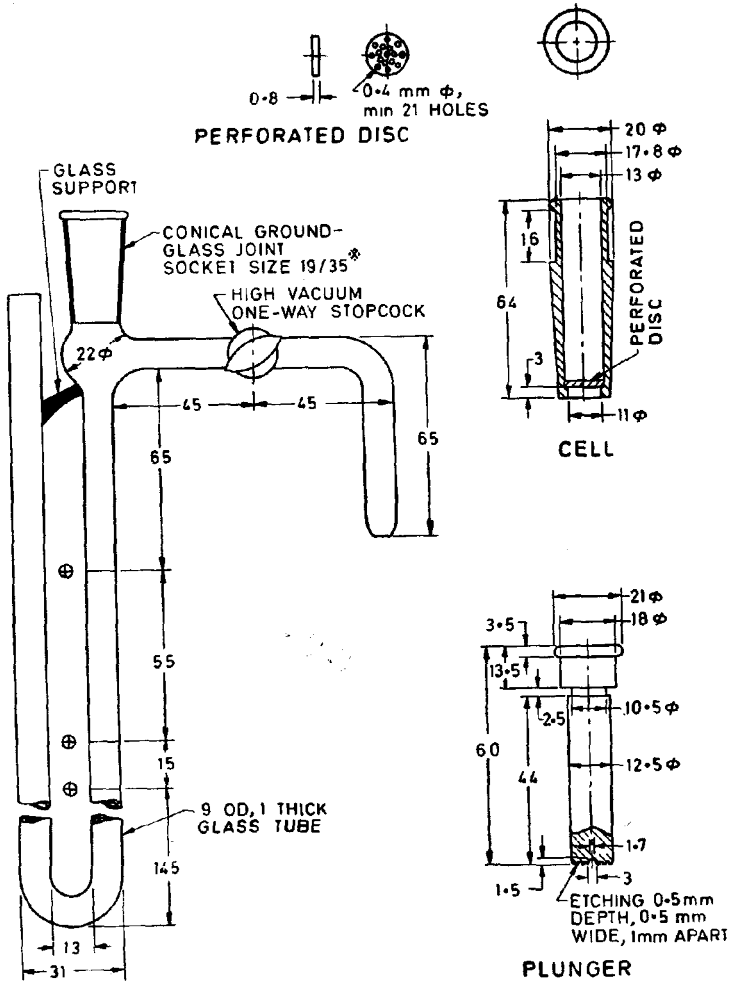
A-5.2.3 Determine the volume of manometer arm between marks B and C by filling from a burette. This volume (V), in millilitres will be equal to the volume of air passing through the bed in the experiment.

*This apparatus is known as Blain air permeability fineness tester.



All dimensions in millimetres.

FIG. 1 APPARATUS FOR THE DETERMINATION OF AVERAGE PARTICLE SIZE — AIR PERMEABILITY FINENESS TESTER



**See* IS : 5165-1969 Interchangeable conical ground-glass joints.

NOTES:

Total height of the tube: Approximate 295 mm.

Tolerance on dimensions:

i) On lengths ± 1 mm

ii) On outer dia and thickness of the tube: ± 0.1 mm

iii) None on joints and marking.

⊕ Circular mark to be etched.

Material: Stainless steel for Cell and Plunger; Brass for Perforated Disc.

All dimensions in millimetres.

FIG 2 DIMENSIONS OF APPARATUS FOR DETERMINATION OF AVERAGE PARTICLE SIZE — AIR PERMEABILITY TESTER

As in the Original Standard, this Page is Intentionally Left Blank

A-5.3 Procedure

A-5.3.1 Mix the whole sample well on a sheet of paper and make sure that its moisture content is within specification. Weigh out exactly 6.240 g of the sample of red lead on to a sheet of glazed paper. The quantity of the material taken is such that it gives a bed of standard porosity of 0.63 when pressed into the test cell (for calculation of porosity, *see* **A-5.4.1**).

A-5.3.2 Place the stainless steel perforated disc in the clean dry stainless steel test cell, making sure that the disc sits properly on the inner ledge.

A-5.3.3 Cut 2 discs of filter paper exactly of the same diameter as the inside of the test cell using the punch provided and place one disc on top of the perforated disc.

A-5.3.4 Place the test cell in the wooden block usually provided with the apparatus and transfer the weighed red lead into the test cell using a funnel.

A-5.3.5 Consolidate and level the powder by tapping the cell. Place the second filter paper disc on top of the powder, insert the stainless steel plunger and push it down until the shoulder of the plunger rests on the lip of the test cell.

A-5.3.6 Withdraw the plunger from the test cell slowly in order to avoid formation of partial vacuum. Fix the test cell firmly into the ground-glass joint.

A-5.3.7 Partially exhaust the air from the arm of the manometer through the glass stopcock and aspirator bulb until the level of the liquid in that arm comes well above the mark *B*.

A-5.3.8 Close the stopcock and note with a stopwatch the time required for the level of the liquid to fall from mark *B* to mark *C*.

A-5.3.9 Repeat the timing operation to give a total of 3 or 5 readings, 3 if the readings are constant and 5 if there is a fair range in the readings.

A-5.3.10 Calculate the average time in seconds required for the liquid to fall from *B* to *C* and determine the specific surface, surface area per unit volume of particles in cm^2/cm^3 and particle size in centimetres as given in **A-5.4.1** and **A-5.4.2**.

A-5.4 Calculation — Calculate the specific surface from the formula given below and report the result to the nearest 100 units as cm^2/cm^3.

A-5.4.1 Specific surface (S), cm^2/cm^3 = $\sqrt{\frac{g\,A\,P\,T}{K\,V\,N\,L} \times \frac{e^3}{(1-e)^2}}$

where

g = acceleration due to gravity in cm/s^2;

A = cross-sectional area of the cell in cm^2 which is the area of bed of powder in cm^2;

T = time of flow of air in seconds;

e = porosity, that is, volume of pore space per unit volume of bed, and is equal to:

$$1 - \frac{(\text{ mass in g of the material })/\text{density of the material}}{\text{volume in ml of the bed}}$$

(density of red lead is 9.1 g/ml and shall be determined for each sample);

K = a constant which has a value of 5;

V = total volume of air passing through the powder in ml and is equal to volume of the manometer arm between marks B and C,

N = viscosity of air in poises. This varies from 0.001 827 poise at 18°C to 0.000 195 8 poise at 40°C and is usually taken as 0.000 185 unless otherwise stated;

L = depth of powder bed in cm; and

P = mean effective pressure difference across the bed of powder, in g per cm^2, and is calculated from the commonly used equation:

$$H = \frac{H_1 - H_2}{\log_e (H_1/H_2)} \quad \ldots\ldots\ldots\ldots (1)$$

H = mean effective head,

H_1 = initial head, and

H_2 = final head.

In terms of the apparatus shown in Fig. 1, the final pressure H_2d = $(CC_1)\ d = h_2d$, where d is the density of the oil in the manometer.

Initial pressure $H_1d = (BC + CC_1 + C_1 B_1)\ d = 2H_1\ d + h_2\ d$.
(B, B_1, C, C_1 and h_2 are same as in Fig. 1)

Substituting these values in equation (1), the mean effective pressure

$$P = \frac{2\ h_1\ d}{\log_e (1 + 2\ h_1/h_2)}$$

or in terms of common logarithm

$$P = \frac{0{\cdot}868\ 6\ h_1\ d}{\log_{10} (1 + 2\ h_1/h_2)}$$

Using diesel oil of density 0.857 3 g/ml at 15°C and a value of 55 mm for h_1 and 30 mm for h_2 as shown in Fig. 1, P works out to 6.12 g/cm^2.

A-5.4.2 *Particle Size* — Calculate the mean particle diameter (size) as follows:

$$\text{Mean particle diameter (in cm)} = \frac{6}{S}$$

where

S = specific surface in cm^2/cm^3 as obtained in **A-5.4.1.**

A-6. ASSAY

A-6.0 Outline of the Method — It depends upon the interaction of lead peroxide and hydrogen peroxide and a titration of the excess of the latter by standard potassium permanganate.

A-6.1 Reagents

A-6.1.1 *Dilute Nitric Acid* — 1:1 (*v/v*).

A-6.1.2 *Dilute Hydrogen Peroxide* — Dissolve 10 ml of 3 percent hydrogen peroxide in 35 ml of water.

A-6.1.3 *Standard Potassium Permanganate Solution* — having an iron value of 0.005. Dissolve 5.75 g of potassium permanganate in 2 litres of water and store in a brown bottle in a dark place for a week or more and filter (*see* Note). In a 400-ml beaker, dissolve 0.25 g of sodium oxalate in 200 to 225 ml of hot water (80 to 90°C) and add 10 ml of dilute sulphuric acid (1 : 1). Titrate the solution at once with potassium permanganate solution, the solution being stirred continuously and vigorously. Add the permanganate at the rate of 10 to 15 ml per minute and the last 0.5 to 1 ml shall be added drop by drop, each drop being allowed to decolourize fully before the next is added. The solution should not be below 60°C by the time the titration is completed. With a permanganate solution having an iron value of 0.005, 41.66 ml of permanganate are required to react with 0.25 g of sodium oxalate. If the titration shows that the solution is too strong, a small amount of water shall be added. To calculate exactly how much water should be added, divide 41.66 by the number of millilitres required in the titration and multiply by the number of millilitres remaining in the bottle. The difference between this product and the number of millilitres remaining in the bottle shall be the volume of water to be added. If the solution is too weak, this difference multiplied by 0.002 83 shall be the grams of potassium permanganate to be added. After addition of water or salt, the solution shall again be titrated and if the titre of 41.66 ml is not obtained, water or salt shall be added until this titre is obtained. The solution carefully prepared in this manner shall be used for titration for a few months.

NOTE — By this time all the organic matter will have been oxidized and after filtering the solution through an asbestos filter, the solution is ready for standardization. As small amounts of manganese dioxide destroy the permanence of this solution, it is necessary that it should be removed by filtering.

A-6.2 Procedure — Take 1 g of the sample in a beaker and add 15 ml of dilute nitric acid (*see* Note). Stir the sample until the red colour has disappeared. Add from a calibrated pipette or burette exactly 10 ml of dilute hydrogen peroxide. Add about 50 ml of hot water and stir until all lead dioxide has dissolved. In the case of some coarsely ground oxides, the contents of the beaker may have to be gently heated to effect complete solution. After the oxides have completely passed into solution, dilute with hot water to about 250 ml and titrate directly with standard potassium permanganate solution having an iron value of 0.005. Titrate to faint pink permanganate colour.

NOTE — The dilute nitric acid shall be aerated to free it from all nitrous fumes before addition.

A-6.2.1 Carry out similarly a blank determination.

A-6.3 Calculation — Calculate the lead as follows:

$$\text{Lead (as } Pb_3O_4 \text{), percent by mass} = 3.069\ (V_1 - V_2)$$

where

V_1 = volume in ml of standard potassium permanganate solution used for the blank titration, and

V_2 = volume in ml of standard potassium permanganate solution used for the red lead titration.

A-7. DETERMINATION OF ARSENIC, ANTIMONY, COPPER, BISMUTH, IRON AND ZINC

A-7.0 Outline of the Method — About 100 to 150 g of the material is treated with dilute nitric acid and hydrogen peroxide, lead is precipitated as $PbSO_4$, filtered and the filtrate concentrated. The trace metallic impurities are separated from each other by the conventional group separation methods and estimated by suitable colorimetric methods.

NOTE 1 — The methods given below are suitable for the small quantities of impurities normally present in red lead and suffice to show whether the limits laid down in Table 1 of the specification have been complied with.

NOTE 2 — It is essential that a blank analysis of all reagents used shall be carried out, along with the analysis of the sample.

A-7.1 Reagents

A-7.1.1 *Dilute Nitric Acid* — 1 : 1 (*v/v*).

A-7.1.2 *Dilute Hydrogen Peroxide* — 3 percent (*m/m*).

A-7.1.3 *Dilute Sulphuric Acid* — 1 : 1 and 1 : 10 (*v/v*).

A-7.1.4 *Concentrated Hydrochloric Acid* — *See* IS : 265-1962*.

A-7.1.5 *Hydrogen Sulphide* — gas.

A-7.1.6 *Ammonium Hydroxide* — 18 N approximately.

A-7.1.7 *Sulphur Dioxide Solution* — saturated.

A-7.1.8 *Potassium Hydroxide Solution* — 10 percent.

A-7.1.9 *Dilute Hydrochloric Acid* — 1 : 1 and 1 : 10 (*v/v*).

A-7.1.10 *Methyl Orange Indicator Solution* — 0.01 percent solution in water.

A-7.1.11 *Concentrated Nitric Acid* — *See* IS : 264-1968†

A-7.1.12 *Ammonium Sulphate Solution* — 2 percent.

A-7.1.13 *Sodium Hydroxide Solution* — 10 percent.

A-7.1.14 *Litmus Indicator Paper*

A-7.1.15 *Sodium Bicarbonate* — solid.

A-7.1.16 *Potassium Iodide Solution* — 2 percent.

A-7.1.17 *Standard Iodine Solution* — 0.01 N.

A-7.1.18 *Starch Indicator Solution* — *See* Sl No. (4), Table VII of IS : 2263-1962‡.

A-7.1.19 *Ammonium Chloride* — solid.

A-7.1.20 *Potassium Thiocyanate Solution* — 10 percent.

A-7.1.21 *Ferric Ammonium Sulphate Solution* — Dissolve 0.086 4 g of ferric ammonium sulphate in water containing 10 ml of concentrated hydrochloric acid and make up to exactly 1 litre in a volumetric flask. One millilitre of this solution is equivalent to 0.01 mg of iron (as Fe).

A-7.1.22 *Dilute Acetic Acid* — 1 : 2 (*v/v*).

A-7.2 Procedure

A-7.2.1 *Separation of Metallic Impurities* — Weigh 100 g of the material and divide it into 4 equal portions. Transfer each portion into a 1 000-ml beaker. Add gradually 300 ml of dilute nitric acid to each portion and stir until the red colour has disappeared. Add 200 to 250 ml of dilute hydrogen peroxide in small quantities to each beaker with gentle heating and stirring all the time till all the red lead has dissolved. Boil the solution

*Specification for hydrochloric acid (*revised*).

†Specification for nitric acid (*first revision*).

‡Methods of preparation of indicator solutions for volumetric analysis.

gently for 10 minutes to decompose the excess of hydrogen peroxide. To the clear solution in each beaker add slowly 50 ml of dilute sulphuric acid (1:1) stirring vigorously. Heat for 5 minutes. Cool and allow the lead sulphate precipitate to settle. Filter the contents of each beaker on one Buchner funnel through a double filter paper and wash the combined precipitate with 300 ml of cold dilute sulphuric acid (1 : 10). Reject the lead sulphate. Treat the filtrate as follows:

Filtrate : Evaporate the combined filtrate in a porcelain dish to 100 ml, rinse into a 250-ml beaker with water making a total bulk of 200 ml. Boil with continuous stirring, cool and filter through a double filter paper and wash with dilute sulphuric acid (1:10) and finally twice with water.

Precipitate: Rinse the paper with a minimum amount of water (about 10 ml) add concentrated hydrochloric acid and boil until the lead sulphate is dissolved. Pour into 20 times its bulk of boiling water, which should give a clear solution. Saturate with H_2S and allow to stand in a warm place for about an hour. Filter and wash once with warm water. Reject the filtrate. Combine this precipitate *'A'* with precipitate *'B'*.

Filtrate: Nearly neutralize with ammonium hydroxide, add 5 ml saturated sulphur dioxide solution and 2 ml sulphuric acid (1:1), total bulk about 100 ml. Allow to stand on a warm plate for 2-3 hours until SO_2 is removed. Cool, saturate with H_2S, allow to stand overnight and filter.

Precipitate 'B' : Combine with precipitate *'A'*. Boil with 5 ml of KOH solution and filter washing the precipitate with water.

Filtrate: contains iron and zinc. Proceed as in **A-7.2.1.3.**

Precipitate: contains lead, bismuth and copper. Proceed as in **A-7.2.1.1.**

Filtrate: contains arsenic and antimony. Proceed as in **A-7.2.1.2.**

A-7.2.1.1 *Determination of bismuth and copper* — Rinse the precipitate off the paper and pour 10 ml hot dilute HNO_3 over the paper into the beaker and wash with hot water. Add sufficient concentrated HNO_3 to dissolve the whole of the sulphide, evaporate until the sulphur globulates, add 5 ml dilute HCl (1:1) and 20 ml of water, boil, filter off the sulphur and wash with dilute HCl (1:10). Dilute filtrate to about 100 ml with hot water, boil to dissolve lead chloride, neutralise with ammonium hydroxide, using methyl orange as indicator, add 2 drops of dilute HCl (1 : 1) in

excess, dilute to 350 ml and allow to stand on a hot-water bath to enable bismuth oxychloride to coagulate. Filter and wash with hot water.

Precipitate — Dissolve the precipitate off the paper with 10 ml HCl (1:1) and 20 ml of dilute H_2SO_4 (1:1) and evaporate to fumes. Cover the beaker and heat until a clear solution is obtained. Cool, dilute with water and transfer to a 250-ml graduated flask. Determine bismuth colorimetrically as prescribed in **A-7.2.2.**

Filtrate — Heat filtrate nearly to boiling and pass H_2S until the solution is quite cold and then filter. Reject the filtrate. Rinse the precipitate off the paper with water into a 200-ml beaker. Pour over the paper 10 to 20 ml of dilute HNO_3, add 20 ml of dilute H_2SO_4 (1 : 1) and evaporate till fumes appear. Cool, dilute with 100 ml of water. Boil and allow to stand overnight. Filter and wash with dilute H_2SO_4 (1 : 10). Reject the precipitate — $PbSO_4$; warm the filtrate and pass H_2S for one minute. Stir the precipitate until the copper precipitate coagulates; then filter quickly washing the precipitate with (1 : 10) H_2SO_4 containing a little H_2S. Ignite the precipitate of CuS at dull red heat. Dissolve in 1 to 2 ml of concentrated HNO_3. Determine copper colorimetrically as prescribed in **A-7.2.3.**

A-7.2.1.2 *Determination of arsenic and antimony* — Evaporate the filtrate obtained after separation of lead, bismuth and copper to 10 ml bulk in a 50-ml beaker and pour into 25 ml concentrated HCl, saturate with H_2S and filter. Wash two or three times with H_2S water acidified with HCl_2 then with ammonium sulphate solution until free from chlorides.

Precipitate — Arsenic — Pour over the paper 3 ml of hot NaOH solution into a 100-ml heat resistant glass flask, containing 3 ml of concentrated H_2SO_4 and wash the paper with hot water. Evaporate to fumes and heat until a colourless solution is obtained. Allow to cool, dilute with 20 ml of cold water, boil to remove any trace of SO_2, cool. Determine arsenic by method (a) or (b), given below.

a) Neutralize with ammonium hydroxide using litmus paper as indicator, make slightly acidic with dilute H_2SO_4 (1 : 10), cool, add excess of $NaHCO_3$, 10 ml of KI solution (2 percent) and titrate with 0.01 N iodine solution, using about 1 ml of freshly prepared starch solution as indicator. (1 ml of 0.01 N iodine = 0.000 3746 g of arsenic).

b) Colorimetric method — as prescribed in **A-7.2.4.**

Filtrate — Neutralize with ammonia, acidify slightly with HCl (1:1) and saturate with H_2S. Filter and with the precipitate with H_2S water. Treat the precipitate to determine antimony as prescribed in **A-7.2.5.**

A-7.2.1.3 *Determination of iron and zinc* — Boil off H_2S from the filtrate obtained after the removal of copper and arsenic group as sulphides;

oxidize iron with bromine water, boil off bromine, add 2 g of ammonium chloride, 2 drops of methyl red solution and ammonium hydroxide drop by drop until the methyl red changes to yellow, warm and filter. Redissolve the precipitate in dilute HCl (1 : 1) and reprecipitate as before. Filter.

Precipitate — Dissolve in 5 ml of dilute HCl (1:1), dilute to 100 ml in a graduated flask and thoroughly mix the solution. Take an aliquot portion of the diluted solution in a 100-ml Nessler cylinder, add 5 ml HCl and 5 ml 10 percent solution of KCNS and dilute up to 100-ml mark. Match the colour with freshly prepared standard ferric ammonium sulphate solution (1 ml = 0.001 mg of Fe, *see* **A-7.1.21**) similarly treated.

Filtrate — Nearly neutralize with HCl, finally with HC1 (1:10) (using methyl orange), add 2 drops of dilute acetic acid (1:2), saturate the hot solution with H_2S, settle overnight and filter. Ignite the precipitate and weigh as zinc oxide. Alternatively, dissolve the ZnS precipitate in 10 ml HCl (1 : 10). Dilute with water, boil to drive off H_2S and make up to 100 ml. Use an aliquot to determine zinc as prescribed in **A-7.2.6.**

A-7.2.2 *Colorimetric Method for Determination of Bismuth*

A-7.2.2.1 *Outline of the method* — When potassium iodide solution is added to dilute sulphuric acid or dilute nitric acid containing a small amount of bismuth, a yellow colouration due to formation of a complex iodide is produced. The colouration is then compared against standards similarly prepared.

A-7.2.2.2 *Reagents*

a) *Standard solution of bismuth* — Dissolve 0.105 g of bismuth in 20 ml of concentrated sulphuric acid and dilute to 1 litre with water. One millilitre of this solution contains 0.1 mg of bismuth (Bi).

b) *Potassium iodide solution* — 10 percent (*m/v*).

c) *Sulphurous acid* — Dilute 1 part of saturated solution of sulphur dioxide with 10 parts of water.

A-7.2.2.3 *Procedure* — Take an aliquot (20-30 ml) of the sample solution in a Nessler cylinder, add 5 ml of potassium iodide solution and 10 ml of sulphurous acid and dilute to mark. Run a blank in another Nessler cylinder without the sample. Into the blank, add enough standard bismuth solution to match the colour produced by the sample. Both the blank and the sample shall have the same concentrations of sulphuric acid and sulphurous acid. Calculate the content of bismuth from the amount of standard bismuth solution consumed.

A-7.2.3 *Colorimetric Method for Determination of Copper*

A-7.2.3.1 *Outline of the method* — An aqueous solution of sodium diethyldithiocarbamate gives with slightly acid or ammoniacal solution of copper salt a brown precipitate of a sparingly soluble copper derivative.

The copper derivative is soluble in organic solvents like chloroform giving a yellowish brown colour which is compared against standard similarly produced.

A-7.2.3.2 *Reagents*

a) *Sodium diethyldithiocarbamate solution* — Dissolve 0.1 g of the reagent in 100 ml of water. Preserve in amber-coloured glass bottle. The solution should not be used after 2 weeks.

b) *Standard copper solution* — Dissolve 0.392 8 g of crystallized copper sulphate ($CuSO_4.5H_2O$) in water. Add enough concentrated sulphuric acid to make the final acidity about 0.1 N and dilute to 1 litre. One millilitre of this solution contains 0.1 mg of copper (as Cu).

c) *Dilute sulphuric acid* — 1 : 10 *(v/v)*.

d) *Citric acid solution* — 10 percent.

e) *Ammonium hydroxide* — 18 N.

f) *Carbon tetrachloride*

g) *Sodium sulphate* — anhydrous.

A-7.2.3.3 *Procedure* — Add 10 ml of water to the copper nitrate solution obtained after separation from all other trace impurities. Boil to expel the excess of nitric acid. Add a drop of dilute sulphuric acid and transfer to a separating funnel. Add 2.5 ml of citric acid solution. Neutralize the excess of acid with ammonium hydroxide and adjust *p*H to 9.0 to 9.2 by addition of ammonium hydroxide. Add 10 ml of 0.1 percent aqueous solution of sodium diethyl dithiocarbamate, mix and immediately extract with 2.5 ml of carbon tetrachloride. Run the lower layer into a dry 20-ml measuring flask. Repeat the extraction with 3 further successive 2.5-ml portions of carbon tetrachloride and combine the extracts. The final 2.5-ml extract should be colourless; if not repeat the extraction until all the complex is removed. Dilute the combined extracts to 20 ml and clarify by adding about 1 g of anhydrous sodium sulphate. Carry out a control test using 20 ml of standard copper solution, proceeding exactly as for the test sample. Compare the intensity of the colour produced in the test and standard samples.

The limit laid down in Table 1 shall be taken as not having been exceeded if the intensity of colour produced with the test sample is not greater than that produced with the standard sample.

A-7.2.4 *Colorimetric Method for Determination of Arsenic*

A-7.2.4.1 *Procedure* — Pipette an aliquot of the solution under test containing 1.0 g of the material and determine arsenic by the Gutzeit method as prescribed in IS : 2088-1971*, using for comparison a stain obtained with

*Methods of determination of arsenic *(first revision)*.

0.005 mg of arsenic trioxide (as As_2O_3). The limit prescribed in Table 1 shall be considered to have not exceeded if the length as well as intensity of the stain produced with the material is not greater than that produced with the standard sample.

A-7.2.5 *Colorimetric Method for Determination of Antimony*

A-7.2.5.1 *Outline of the method* — When aqueous solution of potassium iodide and pyridine are treated with a solution containing small amounts of antimony, a yellow colouration is produced. This colour is compared against colour produced with standard sample similarly treated.

A-7.2.5.2 *Reagent*

Standard solution of antimony — Dissolve 0.266 8 g of potassium antimony tartarate [K (SbO) $C_4H_4O_6$] in 1 000 ml of 10 percent sulphuric acid. One millilitre of this solution is equivalent to 0.1 mg of antimony (Sb).

A-7.2.5.3 *Procedure* — Wash the antimony sulphide precipitate from the filter paper into the beaker in which the precipitation had been effected, treat the paper with 1 or 2 ml of hot dilute sodium hydroxide solution and wash with hot water. Add 5 ml of concentrated sulphuric acid and evaporate to fumes. Just before fuming add one or two drops of concentrated nitric acid to destroy any traces of organic matter derived from the filter paper. Do not fume longer than necessary to dissipate nitric acid. Take up with 15 ml of water. Heat just to boiling and cool.

Measure the following reagents in the order given:

a) 10 ml of 1 percent gum arabic solution
b) 5 ml of 20 percent potassium iodide solution
c) 1 ml of 10 percent aqueous pyridine solution
d) 1 ml of sulphur dioxide solution prepared by 10-fold dilution of a saturated aqueous solution
e) 30 ml of cold dilute sulphuric acid (1:3 *v/v*)

Add the cooled solution of the sample, rinse it with a further 30 ml of dilute sulphuric acid (1 : 10) and make up to 100-ml mark.

Measure the same amounts of reagents as above into a second Nessler cylinder and increase the quantity of dilute sulphuric acid (1 : 10) to 80 ml instead of 60 ml. Add known volume of standard antimony solution until the yellow colour produced matches that of the sample. Calculate the content of antimony from the amount of standard solution consumed.

A-7.2.6 *Colorimetric Method for Determination of Zinc*

A-7.2.6.1 *Reagents*

a) *Standard solution of zinc*

i) *Stock solution* — Dissolve 1.000 g of zinc metal in 100 ml of 0.1 N hydrochloric acid, make up to one litre with water, and mix the solution well. One millilitre of this solution is equivalent to 1 mg of zinc (Zn).

ii) *Standard solution* — Dilute 10 ml of the stock solution with sufficient water to make exactly one litre with water, and mix the solution well. One millilitre of this solution is equivalent to 0.01 mg of zinc (Zn). The standard solution should be prepared afresh when required.

b) *Dithizone solution* — 0.001 percent in carbon tetrachloride, prepared from purified dithizone. Purify dithizone as given below:

Dissolve 1 g of dithizone in 75 ml of chloroform, filter and shake the filtered solution, contained in a 250-ml separating funnel, with four successive 100-ml portions of approximately 0.2 N sodium hydroxide solution. Combine the orange-coloured aqueous extracts, filter into a 1-litre beaker, and precipitate the dithizone by slightly acidifying the solution with saturated sulphur dioxide water. Alternatively, sulphuric acid (3 *M*) may be used to precipitate the dithizone, but the product obtained is then not quite so pure or stable. After settling, filter off the precipitate on a sintered glass crucible, using gentle suction, and wash it acid-free with water. Dry the well-drained precipitate over concentrated sulphuric acid *in vacuum* for 3 to 4 days, protecting it from light. Grind the solid rapidly and lightly, and transfer it immediately to a small amber-coloured bottle. The purified solid, so prepared, when stored in the dark is stable for at least six months.

c) *Acetate buffer, p*H *about* 4.75 — Extract 500 ml of 2 N ammonium acetate with 0.01 percent dithizone solution till the former is free from heavy metals. Mix the resulting solution with 500 ml of 2 N acetic acid.

d) *Sodium thiosulphate solution* — Dissolve 25 g of sodium thiosulphate ($Na_2S_2O_3.5H_2O$) in 100 ml of water and extract the solution with 0.01 percent dithizone solution until it is free from heavy metals.

A-7.2.6.2 *Preparation and measurement of optical density of coloured solution* — Measure 0.1, 0.3, 0.5, 0.7 and 1.0 ml lots of the standard zinc solution into 50-ml separating funnels and make the volume of each solution to 10 ml with water. Add two drops of bromocresol green indicator and ammonium hydroxide (10 *N*) till the solution just turns yellowish-green. Add 10 ml of the acetate buffer and 1 ml of the sodium thiosulphate solution. Shake for 2 minutes with 20 ml of dithizone solution. Dry the inside of the stem of the separating funnel with filter

paper and run the dithizone layer into a 4-cm cell. Prepare a reagent blank in the same manner.

Measure the optical density of the solution with reference to the reagent blank at a wavelength of 520 nm using a calibrated absorptiometer. Make at least three independent determinations for each concentration and plot the calibration curve with concentrations as ordinates and optical density as abscissae (the points should lie on a straight line passing through, or nearly through, the origin).

A-7.2.6.3 *Periodic checking* — Make two of the standard solutions described in **A-7.2.6.2** and measure their optical density as directed; carry out a check of this type at least once every three months.

A-7.2.6.4 Pipette a suitable aliquot of the sample solution into a 50-ml separating funnel and proceed as directed in the preparation of the calibration curve for zinc. At the same time prepare a reagent blank omitting only the sample. Make the necessary correction for the reagent blank and calculate the amount of zinc present in the original sample.

APPENDIX B

(*Clause* 4.1)

SAMPLING OF RED LEAD FOR EXPLOSIVE AND PYROTECHNIC INDUSTRY

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Samples shall not be taken in an exposed place.

B-1.2 The sampling instrument and the containers for samples shall be made of metal or opaque glass on which the material has no action. They shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 The sample containers shall be of such a size that they are almost, but not completely, filled with the sample.

B-1.5 The sample containers shall be sealed air-tight after filling and marked with full identification particulars, such as the date of sampling,

month and year of manufacture of the material, the name of the sample and any other relevant particulars of the consignment.

B-1.6 Samples shall be stored in such a manner as to avoid excessive variations of temperature.

B-2. SCALE OF SAMPLING

B-2.1 Lot — In a single consignment of the material all the containers of the same size and drawn from the same batch of manufacture shall constitute a lot. If a consignment is known to consist of different sizes of containers, the containers belonging to the same batch and size shall be grouped together and each such group shall constitute a separate lot.

B-2.1.1 For ascertaining the conformity of the material in a lot to the requirements of the specification, tests shall be carried out for each lot separately. The number of containers to be selected at random from lots of different sizes shall be in accordance with Table 2.

TABLE 2 SCALE OF SAMPLING

LOT SIZE *N*	SAMPLE SIZE *n*
(1)	(2)
Up to 20	1
21 to 60	2
61 to 150	3
151 to 300	4
301 to 500	5
501 and above	6

B-2.1.2 These containers shall be selected at random. In order to ensure the randomness of selection, random number tables given in IS : 4905-1968* may be used. In case such tables are not available, the following procedure may be adopted:

Starting from any container, count them in one order as 1, 2, 3, ... up to r and so on, where r is the integral part of N/n. Every rth container thus counted shall be taken out until the sample of required size is obtained.

*Methods for random sampling.

B-3. PREPARATION OF TEST SAMPLES

B-3.1 From each of the containers selected, draw with an appropriate sampling instrument a small representative portion of the material approximately 100 g in mass.

B-3.2 Out of these portions, small but equal quantity of the material shall be taken and mixed thoroughly to form a composite sample of mass about 200 g. This composite sample shall be divided into three equal parts, one for the purchaser, another for supplier and the third to be used as a referee sample.

B-3.3 The referee sample shall bear the seal of both the purchaser and the supplier. It shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of dispute between the two.

B-4. NUMBER OF TESTS

B-4.1 Tests for the determination of all the characteristics mentioned in Table 1 shall be carried out on the composite sample.

B-5. CRITERIA FOR CONFORMITY

B-5.1 For declaring the conformity of the lot to the requirements of this specification, the test results for each of the characteristics shall meet the corresponding values specified in Table 1.

INDIAN STANDARDS

ON

EXPLOSIVES AND RAW MATERIALS FOR EXPLOSIVES

IS:

301-1963 Potassium nitrate for explosives and pyrotechnic compositions (*revised*)
438-1972 Aluminium powder for explosives and pyrotechnic compositions (*first revision*)
708-1970 Potassium chlorate, technical (*first revision*)
2012-1961 Red phosphorus
2307-1962 Magnesium powder for explosives and pyrotechnic compositions
4396-1967 Barium nitrate for explosives and pyrotechnic compositions
4668-1967 Ammonium nitrate for explosives
5670-1970 Lead thiocyanate for explosive and pyrotechnic composition
5671-1970 Strontium nitrate for pyrotechnic compositions
5713-1970 Manganese dioxide for explosive and pyrotechnic compositions
5731-1970 Antimony sulphide for explosive and pyrotechnic compositions
6609 (Part I)-1972 Methods for test for commercial blasting explosives and accessories : Part I Gun powder
6609 (Part II/Sec 1)-1973 Methods of test for commercial blasting explosives and accessories : Part II Explosives; Section 1 Explosives, general
6609 (Part II/Sec 2)-1974 Methods of test for commercial blasting explosives and accessories : Part II Explosives; Section 2 Explosives, permitted
6609 (Part III)-1973 Methods of test for commercial blasting explosives and accessories : Part III Detonators, general and permitted
6609 (Part IV)-1972 Methods of test for commercial blasting explosives and accessories : Part IV Detonating fuses
6609 (Part V)-1972 Methods of test for commercial blasting explosives and accessories : Part V Safety fuses
7124-1973 Gun powder
7401-1974 Paraffin wax for explosive and pyrotechnic industry
7526-1974 Detonating fuses
7588-1974 Barytes for explosive and pyrotechnic industry
7589-1974 China clay for explosive and pyrotechnic industry
7602-1975 Lead chromate for explosive and pyrotechnic compositions
7632-1975 Detonators
7633-1975 Calcium carbonate for explosives and pyrotechnic industry
7738-1975 Safety fuse for commercial use
7886-1975 Barium chromate for explosive and pyrotechnic industry

INDIAN STANDARDS INSTITUTION

Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002

Telephone : 27 01 31 (20 lines) Telegrams : Manaksanstha

Regional Offices:

			Telephone
Western :	Novelty Chambers, Grant Road	BOMBAY 400007	37 97 29
Eastern :	5 Chowringhee Approach	CALCUTTA 700072	23 08 02
Southern :	C.I.T. Campus, Adyar	MADRAS 600020	41 24 42

Branch Offices:

'Pushpak', Nurmohamed Shaikh Marg, Khanpur	AHMADABAD 380001	2 03 91
'F' Block, Unity Bldg, Narasimharaja Square	BANGALORE 560002	2 76 49
Ahimsa Bldg, SCO 82-83, Sector 17C	CHANDIGARH 160017	2 83 20
5-8-56/57 Nampally Station Road	HYDERABAD 500001	4 57 11
117/418 B Sarvodaya Nagar	KANPUR 208005	82 72
B.C.I. Bldg (3rd Floor), Gandhi Maidan East	PATNA 800004	5 36 55
Hantex Bldg (2nd Floor), Rly Station Road	TRIVANDRUM 695001	32 27

Printed at Delhi Printers, Delhi, India